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DENSITY OF HYDROXYLAMMONIUM NITRATE SOLUTIONS

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DECEMBER 1988

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DENSITY OF HYDROXYLAMMONIUM NITRATE SOLUTIONS

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Hydroxylammonium nitrate is miscible with water in all proportions at room temperature. The densities of such salt solutions were measured using water and deuterium oxide as solvents. A second order equation is given to relate density to molar concentration.

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I. INTRODUCTION

Through the long term Liquid Propellant Program at the BRL, there was a need to measure the density of ingredients for one or more purposes and approximate measurements were made to satisfy various requirements as they existed at particular times. Measurements were made at varying room temperatures during a particular series of days and this generated some useful data as opposed to conducting a careful and systematic study. Sasse'[1] in 1976 used an approximate equation for the density, ρ, in g/cm^3, of hydroxylammonium nitrate solutions, HAN, derived from data from 1 to 13 M. The equation was:

$$\rho = 1.0000 + 0.0369\ M \qquad (1)$$

He made the suggestion that as a first approximation, the ingredients of HAN based liquid propellants when added one to another acted as independent volume elements much like "marbles". In this approximation the density of pure HAN was inferred, by experiments, to be 1.81 g/cm^3 and this value was used later by Freedman and Travis[2] to estimate propellant concentrations and densities as input to thermodynamic calculations.

The first disclosure that the density/concentration relationship may not be linear was offered by Biddle[3] who related hydrometer readings to concentration. In this work two hydrometers of different radii were used and surface tension corrections were not made; also, these measurements were conducted at the room temperature experienced at particular days. Such readings were compiled as a practical expedient for establishing production guides and criteria for maintaining and evaluating evaporation methodology. He showed a single regression line, but not the individual data points, embracing all data from 2.1 to 17.3 M HAN. The equation was given as:

$$M = 26.04\rho - 26.38 \qquad (2)$$

which can be transformed to:

$$\rho = 1.013 + 0.0384\ M \qquad (3)$$

To meet the production goal of 13 M he also made and graphed extensive hydrometer measurements representing the very limited concentration interval of 11.7 to 14.3 M. He presented these values in tabular form in units of percent concentration. Further, he assumed that he could represent the percent concentration/density data, which is a curving function, as a limited linear segment, and gave an equation for that regression line. From his table of percent concentrations and densities, molarity can be calculated. A linear regression equation of this data was uniquely reported in a physical properties paper[4] over this limited range as:

$$\rho = 1.12292 + 0.03099\ M \qquad (4)$$

This line is given in Figure 1 of this paper together with Biddle's regression line representing all of his data from 2.1 to 17.3 M. The discontinuity between the treatment of the entire data set and an extracted subset, indicates that the density/concentration relationship may be curved; however, from the limited range of the data in the subset and noting the uncertainties involved, it is not clear that the purported curvature is significant. Biddle

used the equivalent of Eq. 4, but in units of percent concentration, in his evaporation task where the approximations involved did not effect his objective. However, he did not explore or discuss the implied curvature.

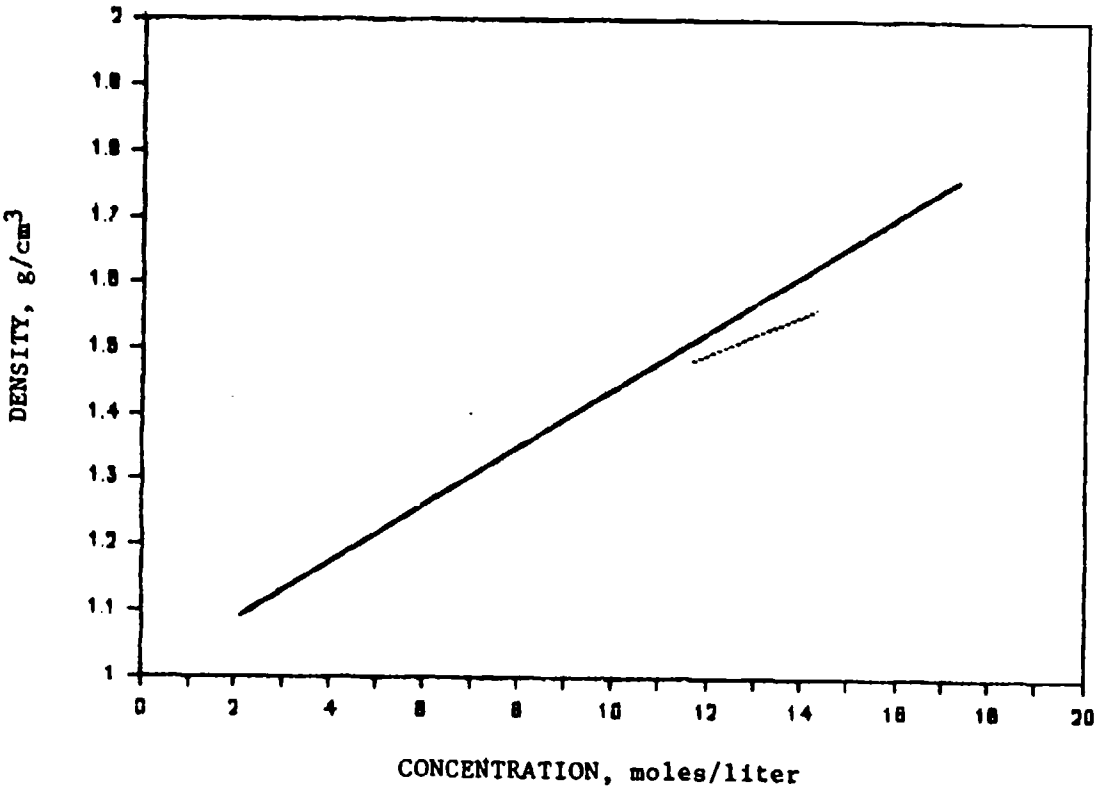

Figure 1. Biddle's Data Reference 3. Upper line is the linear regression line of all data and lower line is the linear regression line of a limited interval.

With the acquisition of temperature controlled density equipment* at the BRL, based on the measurement of the frequency of an oscillator, and the ability to make pure crystalline HAN (which was used to make concentrated solutions), encouraged us to re-examine the relationship between density and concentration. Further persuasion was offered by the fact that Decker of the BRL was routinely analyzing HAN solutions and Davies was preparing deuterated HAN (noted as dHAN) in D_2O for his diamond anvil cell infrared studies.[5] Also, Rheingold, et al.,[6] determined the density of pure crystalline HAN and dHAN by x-ray methods and Sasse' and Fifer measured the density of pure liquid HAN and dHAN with a pycnometer. Measurements are given in Table 1 and plotted in Figure 2. Decker titrated all HAN and dHAN solutions with strong base and determined the densities of HAN solutions while Davies measured densities of dHAN solutions made with D_2O. Both experimenters operated at 20.0°C. From measuring the density of pure water, the density determinations are judged to be accurate to about 0.14% and the HAN analysis has a precision of 4-5 parts per thousand.

*Mettler/Parr Density Meter, Model DMA 55

Table 1. Density of HAN and dHAN and Their Solutions

HAN		dHAN	
Density g/cm^3	Concentration M	Density g/cm^3	Concentration M
0.99963	0.0-pure H_2O	1.1053	0.0-pure D_2O
1.02341	0.500	1.1662	1.336
1.12533	2.781	1.1904	1.875
1.22919	5.232	1.2761	3.936
1.29636	6.858	1.4488	8.321
1.37548	8.839	1.6137	12.74
1.52306	12.622	1.69694	15.86*
1.65569	16.338		
1.68	17.5**	1.841	18.40**
1.841	19.17***	1.916	19.15***

* only value calculated by weight.
** anhydrous melt.
*** crystalline value, Reference 6.

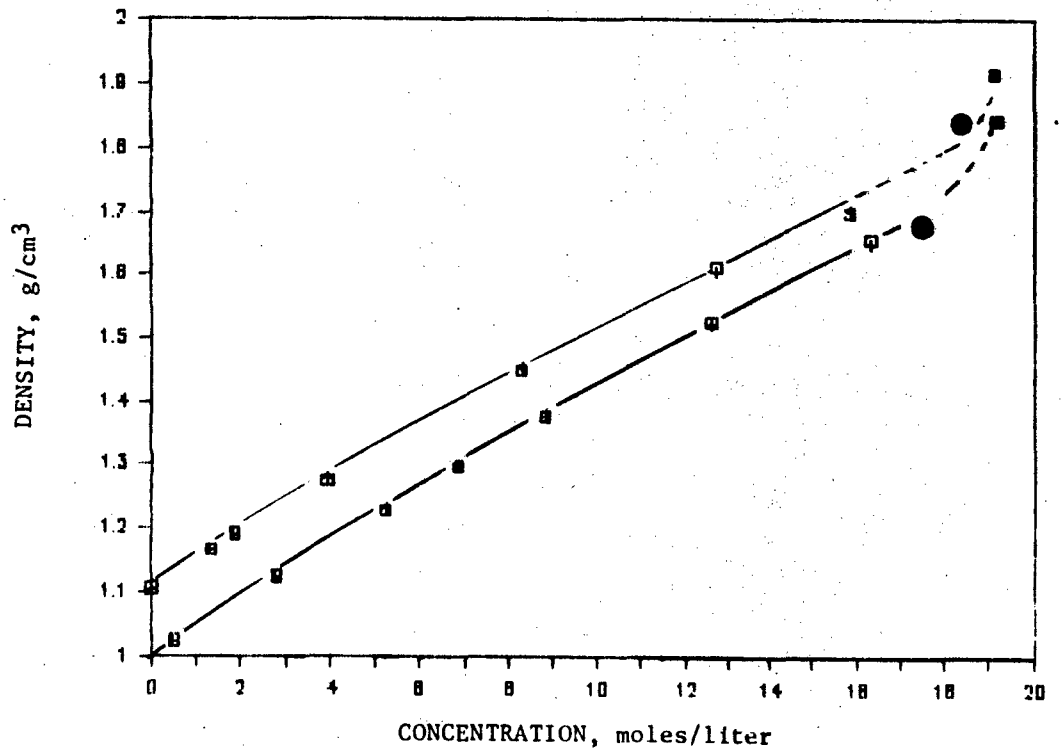

Figure 2. Second Order Regression Lines for dHAN and HAN Systems. Upper line is dHAN and lower line is HAN data. Squares are experimental data and crosses are calculated regression values. Filled in squares are crystalline values and filled in circles are pure melted compounds.

II. DISCUSSION

Recognizing the constraints, limitations, and concentration ranges employed by various investigators the collection of density relationships are all in general agreement. However it is hoped that the present data set reflects the effort of careful temperature control and the curvature noted in Figure 2 is reinforced by the availability of data at high concentrations. Second order least squares and linear regression analyses were performed by Kotlar using the data in Table 1 excluding crystalline values. Statistical details are given in the Appendix showing that second order equations better represent the data and the best fit equations are given below:

$$\rho_{HAN} = 0.9935 + 0.04630\ M - 0.0004007\ M^2 \text{ in } H_2O \qquad (5)$$

$$\rho_{dHAN} = 1.1045 + 0.04646\ M - 0.0005581\ M^2 \text{ in } D_2O \qquad (6)$$

One would expect that the curvature noted by the above equations would also be present in refractive index functions, a parameter that some of us wish to document.

Some structural information may be deduced by including the crystalline and anhydrous liquid HAN values in the concentration plot given in Figure 1. For instance, if one considers the melting of an ionic crystal, then the melt contains the maximum number of ion pairs possible, what ever that number might be. In the present case, HAN changes density from 1.841 to 1.68 upon melting and with the addition of increments of water one would expect the number of ion pairs to decrease until a dilute solution is achieved and only solvation takes place. If the observed curvature in the density plot is due to such effects, then ion pairs have a dominant effect upon this parameter from saturated solutions to ca. 8 M. Carrying this concept further, then dilute solutions would not show ion pairing but just free ions would exist and extrapolation of these data, 0 to 8 M, should intersect the crystalline density value. This is indeed the case, and such an extrapolation may be made in Figure 2 for both the HAN and dHAN data sets. The figure is too busy to make this construction and this task is left to the reader. The argument, if correct, implies that the ion pairs formed would have a greater volume than the sum of the volumes of its constituents.

It is the general conclusion of this report that the functional relationship between density and molar concentration of HAN solutions, over its entire concentration range, is represented by a second order equation that is very similar to the equation for the deuterated system.

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6. A.L. Rheingold, J.T. Cronin, T.B. Brill, and F.K. Ross, "Structure of Hydroxylammonium Nitrate (HAN) and the Deuterium Homolog," Acta Cryst., Vol. C43, pp. 402-404, 1987.

APPENDIX

A linear regression line and a second order regression fit was performed on both data sets , HAN and dHAN, given in Table 1. Concentration is in units of moles/liter(M) and density (ρ) is in units of grams/cubic centimeter. Results are:

Table 2. Regression Analyses

Coefficient	Standard Deviation
HAN for: $\rho = P1 + P2\ (M)$	
P1 = 1.0145	7.690E-3
P2 = 3.9301E-2	7.720E-4
Density	1.4195E-2
HAN for: $\rho = P1 + P2\ (M) + P3\ (M^2)$	
P1 = 0.9935	2.983E-3
P2 = 4.6297E-2	8.771E-4
P3 =-4.0066E-4	4.842E-5
Density	4.3519E-3
dHAN for: $\rho = P1 + P2\ (M)$	
P1 = 1.1191	8.506E-3
P2 = 3.7755E-2	1.003E-3
Density	1.5086E-2
dHAN for: $\rho = P1 + P2\ (M) + P3\ (M^2)$	
P1 = 1.1045	3.622E-3
P2 = 4.6458E-2	1.392E-3
P3 =-5.5811E-4	8.670E-5
Density	5.0046E-3

It is interesting that the statistics for both the HAN and dHAN data are comparable and thus the experiments are of equal percision. This is the desired result; however, due to "improvements" such an occurrence is not seen too often.

The standard deviation for density using the quadratic equation is three times smaller than given by the linear equation; therefore, the data for HAN and dHAN are better represented by the quadratic equations given above. These equations are reproduced in the body of the text.

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This Laboratory undertakes a continuing effort to improve the quality of the reports it publishes. Your comments/answers to the items/questions below will aid us in our efforts.

1. BRL Report Number _____ Date of Report _____

2. Date Report Received _____

3. Does this report satisfy a need? (Comment on purpose, related project, or other area of interest for which the report will be used.) _____

4. How specifically, is the report being used? (Information source, design data, procedure, source of ideas, etc.) _____

5. Has the information in this report led to any quantitative savings as far as man-hours or dollars saved, operating costs avoided or efficiencies achieved, etc? If so, please elaborate. _____

6. General Comments. What do you think should be changed to improve future reports? (Indicate changes to organization, technical content, format, etc.)

_____

_____

_____

CURRENT ADDRESS

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Name

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Organization

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Address

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City, State, Zip

7. If indicating a Change of Address or Address Correction, please provide the New or Correct Address in Block 6 above and the Old or Incorrect address below.

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_____
Organization

_____
Address

_____
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(Remove this sheet, fold as indicated, staple or tape closed, and mail.)